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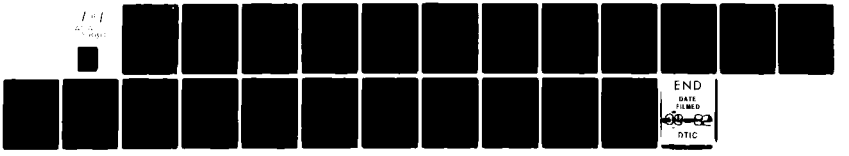

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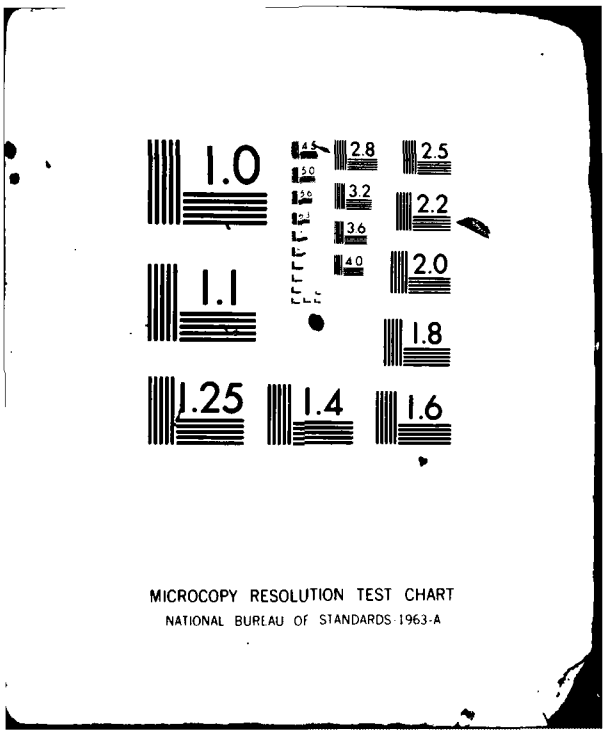
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ANOMALOUS REORGANIZATION FREE ENERGIES IN OPTICAL ELECTRON TRANSFER IN SOLUTION

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Paul Delahay

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ANOMALOUS REORGANIZATION FREE ENERGIES IN OPTICAL ELECTRON TRANSFER IN SOLUTION

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Autoionization bands are observed in the photoelectron emission spectroscopy of aqueous solutions of cyanometalate complexes (Mn, Fe, W, Mo), anions (NO_3^-, ClO_4^-) and cations (Ag^+, Tl^+). Reorganization free energies for autoionization bands are anomalously low in absolute value (by ca. 1 eV) in comparison with direct transitions to the continuum. Interpretation is based on potential energy profiles and model calculations for the reorganization free energy.

Solutions of certain ions (e.g., $Fe(CN)_6^{4-}$) in various solvents exhibit two distinct bands for emission of photoelectrons in the solvent vapor above the solution. The emission bands consist of a plot of the emission yield (number of collected electrons per incident photon) against photon energy. The first band is observed at photon energies lower by ca. 1 eV than expected from the energetics of electron emission and subsequent reorganization of nuclear configurations. Conversely, the second band is observed in the expected range of photon energies. These results will be interpreted in the present paper by considering emission by autoionization and direct transition to the continuum.

1. Autoionization in solution

Photoelectron emission by solutions will be interpreted on the basis of potential energy profiles similar to those used in the discussion of thermal electron transfer [1,2]. The species being photoionized and the resulting product are denoted, respectively, by D^- and D regardless of actual ionic

charges (to simplify notations). The curve $U(D^-)$ in Fig. 1 (left) represents the variations of the potential energy of the solvated species D^- with a generalized coordinate depending on the nuclear configuration of D^- and the solvent. The reduction of potential energy hypersurfaces to a two-dimensional representation is discussed in [1,2]. Curve $U(D,e^-)$ pertains to the products of photoionization, namely D and the electron in the gas phase. The constant potential energy of the electron at infinity in the gas phase is included in $U(D,e^-)$. The minimum of the $U(D^-)$ curve corresponds to the ground state of D^-. Conversely, the minimum of the $U(D,e^-)$ curve pertains to the ground state of D and the electron at infinity in the gas phase (treated as a vacuum). The two minima are separated by the energy gap U_c.

Photoelectron emission into the gas phase results from the vertical transition AB (Franck-Condon principle) involving the difference in potential energy $U_{AB} = U_c - U_r$, where U_r (< 0) is the energy for reorganization of the solvated species produced by photoionization. The reorganization process is represented by the segment BC of the $U(D,e^-)$ curve. The kinetic energy of the electron emitted into the gas phase depends on U_{AB} and the photon energy.

Suppose that the excited state D^-_{ex} is prepared by the transition AE requiring the change in potential energy U_{AE} (Fig. 1, right). This transition satisfies the condition $U_c < U_{AE} < U_c - U_r$. In that case, radiationless transitions may occur between the discrete levels of D^-_{ex} and continuum levels of (D,e^-), and autoionization may occur [3]. The discrete-continuum interaction can be interpreted as the result of the intersection [1,2] of the potential energy curves of D^-_{ex} and (D,e^-) (Fig. 1, right). If there is autoionization, photoelectron emission into the gas phase is observed for a change of potential energy U_{AE} lower than $U_{AB} = U_c - U_r$. Two distinct emission bands are observed if U_{AB} and U_{AE} differ sufficiently. Moreover, the reorganization

energy $U_r' = U_c - U_{AE}$ for the autoionization band is lower in absolute value than $U_r = U_c - U_{AB}$ for the band for direct transition to the continuum.

Interaction between discrete and continuum levels considered in the preceding discussion was already invoked in the interpretation of the emission bands of anthracene mono- and divalent anion radicals in solution [4]. Autoionization was inferred in that work from the near coincidence of emission and absorption bands. The interpretation was not cast in terms of potential energy profiles, and the reorganization process was not considered. The main points of the present paper therefore did not appear in [4].

2. Anomalous reorganization free energies

Autoionization is detected in this work from the anomalously low absolute value of the reorganization free energy R. Basic equations and methods for the analysis of data were recently reviewed [5]. The value of R is computed from [1,6]

$$\Delta G^m = \Delta G_H + \Delta G - R, \qquad (1)$$

where ΔG^m is the free energy for emission; $\Delta G_H = 4.50$ eV on the assumption of a negligible (±0.1 V) surface potential at the solution-water vapor interface; ΔG is the free energy change for the reaction, $D^-(aq) + H^+(aq) = D(aq) + 1/2H_2(g)$. The sum $\Delta G_H + \Delta G$ in (1) corresponds to the change of potential energy U_c in sec. 1. One sets $\Delta G^m = E^t$, where E^t is the experimentally determined threshold energy. This equality holds within ca. ±0.2 eV or better [7]. The quantity E^t is obtained from a linear plot of Y^n vs. E, where Y is the emission yield, E the photon energy, and n = 0.4 or 0.5. The extrapolation method is discussed in [5].

Anomalously low values of |R| computed from (1) can be identified by comparison with the results of model calculations [1,8,9]. The quantity R is equated to the sum of inner- and outer-sphere contributions denoted by R(in) and R(out), respectively. One has [8],

$$R(out) = -(\varepsilon_{op}^{-1} - \varepsilon_s^{-1})e^2/2a, \qquad (2)$$

where ε_{op} and ε_s are the optical and static dielectric constants of water, respectively, and a is the radius of the boundary between inner- and outer-sphere regions. Equation (2) yields approximate results, but this does not matter for our purpose because of the large difference between normal and anomalous values of R. It is not necessary for the same reason to calculate R(in) in most cases. Equation (2) is applied in sec. 3, and another method for identifying anomalous values of R is discussed in sec. 4.

3. Cyanometalate complexes

Some cyanometalate complexes undergo autoionization. Thus, $Fe(CN)_6^{4-}$ exhibits two distinct emission bands [10], and the values of $|R|$ for the complexes of Table 1 are anomalously low except for the second bands of $Mn(CN)_6^{4-}$ and $Fe(CN)_6^{4-}$. In fact, $|R|$ is amazingly low (0.3 eV) for $W(CN)_8^{4-}$, for instance. The threshold energies in Table 1 were determined from the emission spectra in [11] by application of the extrapolation method used in [6,7] and reviewed in [5]. Data on ΔG are from [12]. One has $a \approx 0.45$ nm [13] for the complexes of Table 1. The corresponding free energy, $R(out) \approx -0.9$ eV, computed from (2) is higher in absolute value than the experimental values of $|R|$, except for the second bands of $Mn(CN)_6^{4-}$ and $Fe(CN)_6^{4-}$. The discrepancy is even greater because R(in) (= -0.38 eV for $Fe(CN)_6^{4-}$ [13]) should be taken into account and eq. (2) yields somewhat low values of $|R(out)|$ [14]. Thus, only the second bands of $Mn(CN)_6^{4-}$ and $Fe(CN)_6^{4-}$ correspond to normal values of R and are assigned, on that basis, to direct transitions to the continuum. The assignment of the second band of $Fe(CN)_6^{4-}$ to autoionization in [10] therefore must be revised.

Autoionization occurs in the range of photon energies in which intense absorption bands are observed [15,16], as required by the analysis of sec. 1. The threshold energies of the autoionization bands of $Mn(CN)_6^{4-}$ (4.8 eV)

and $Fe(CN)_6^{4-}$ (5.5 eV) are slightly lower than the photon energies E^{max} = 4.90 and 5.69 eV [15], respectively, of broad, intense (ϵ^{max} = 31,100 and 24,200 M^{-1} cm^{-1}) charge transfer absorption bands of these complexes. The complexes $W(CN)_8^{4-}$ and $Mo(CN)_8^{4-}$ exhibit intense charge transfer bands [16] at 4.98 eV (ϵ^{max} = 25,060 M^{-1} cm^{-1}) and 5.17 eV (ϵ^{max} = 15,540 M^{-1} cm^{-1}), respectively. These values of E^{max} are lower than the corresponding threshold energies (by 0.4 eV) and the sums $\Delta G_H + \Delta G$ of (1) (by ca. 0.1 eV). This is surprising, and it is possible that these complexes exhibit another absorption band at higher photon energies than the preceding E^{max} values. It is also possible that the E^{max} energies are too low by a few tenths of electronvolt because of the uncertainty in the resolution of the absorption spectrum by superposed multiple Gaussians.

The two emission bands of $Mn(CN)_6^{4-}$ in [11] are not as well resolved as for $Fe(CN)_6^{4-}$ [10], but they are still apparent. The second band of $Mn(CN)_6^{4-}$ has a threshold energy of ca. 6 eV and a normal value $R \approx -1.7$ eV. More exact analysis is not feasible because of the possibility of spurious emission by $Mn(CN)_6^{3-}$ for the easily oxidized (by air) solutions of $Mn(CN)_6^{4-}$. The emission spectra of the other complexes of Table 1 were not investigated in [11] at sufficiently high photon energies (up to ca. 8 eV) to allow the detection of their normal emission bands by the extrapolation method of sec. 4.

4. Inorganic anions and cations

Definite evidence for autoionization was found for NO_3^- and ClO_4^- among the common inorganic anions investigated in [17]. The reorganization free energy of NO_3^- in aqueous solution is anomalously low in absolute value: E^t = 7.46 eV [17], $\Delta G = 2.3 \pm 0.1$ eV [18], $R \approx -0.7$ eV from (1). A second emission band therefore is expected at photon energies corresponding to the

normal value of R. The plot of the emission yield Y to the power 0.5 against photon energy (sec. 2, [5]) does exhibit a break at ca. 9.2 eV for the data obtained in [17]. The threshold energies from the two linear segments (7.9 to 9.1 eV and 9.3 to 10.3 eV) of the extrapolation plot are 7.66 and 8.38 eV, respectively. Extrapolation from a plot of $Y^{0.4}$ rather than $Y^{0.5}$ generally yields better fits for $E^t < 8$ eV [6,7,17] (cf. discussion in [5]). The resulting first threshold energy is 7.47 eV in good agreement with $E^t = 7.46$ eV in [17]. The value R = -1.6 eV computed from (1) for $E^t = 8.38$ eV is normal in contrast with the anomalous value, R = -0.7 eV, for the autoionization band. It should be noted that extrapolation plots of $Y^{0.4}$ or $Y^{0.5}$ against photon energy can yield only an approximate threshold energy of the autoionization band. These plots presuppose a constant product of the emission cross section by the attenuation length of the electron wave in the liquid (cf. discussion in [5]). This can hardly be the case for autoionization bands which have the Gaussian-like shape of absorption bands [4,10].

The change of free energy ΔG is not available for application of (1) to emission by ClO_4^-. The reorganization free energy R can be computed in that case from,

$$E^t = A - \Delta G^S - R, \tag{3}$$

where A is the electron affinity of the ClO_4 radical and ΔG^S the free energy of solvation of ClO_4^-. Equation (3) involves minor approximations which are stated in its derivation in [5]. One computes $\Delta G^S = -2.25$ eV for ClO_4^- from data in [19,20]. One has: $E^t = 8.45$ eV for ClO_4^- [17], A = 5.82 eV [21], and the anomalous value R = -0.38 eV. Thus, the band at $E^t = 8.45$ eV is assigned to autoionization. The validity of this assignment rests, of course, on the corrections of the numerical value of A. A normal value of R would require A to be lower by ca. 1 eV than the value used here.

The cations Ag^+ and Tl^+ among those studied in [6] have anomalous reorganization free energies: $E^t = 7.52$ eV, $\Delta G = 2.0$ eV [12], $R \approx -1.0$ eV for Ag^+; $E^t = 7.40$ eV, $\Delta G = 2.2$ eV [22], $R \approx -0.7$ eV for Tl^+. The anomalous character of these values of R can be ascertained from the ratio $\rho = z^2R/\Delta G^s$, where ΔG^s is the free energy of solvation of the ion having the ionic charge z^+ or $z-$. A numerical value of ρ is obtained from (2) and the Born equation for ΔG^s by neglecting the inner-sphere contribution to R. Thus,

$$\rho \sim (\varepsilon_{op}^{-1} - \varepsilon_s^{-1})/(1 - \varepsilon_s^{-1}). \qquad (4)$$

The term z^2 appears in the ratio $z^2R/\Delta G^s$ because ΔG^s is proportional to z^2 according to the Born equation whereas R(out) of (2) is independent of z. Equation (3) is based on a crude model but it yields the value, $\rho = 0.56$, for aqueous solutions at room temperature in fair agreement with experiment (Table 2), namely $0.42 \leqslant z^2R/\Delta G^s \leqslant 0.53$. It should be noted that the values of ΔG^s used in the calculation of ρ and the quantity ΔG_H in (1) are not independent, as one can ascertain from appropriate thermodynamic cycles. The value of ρ therefore depends somewhat on the choice of the numerical value of ΔG_H in (1). This dependence is not significant for our present purpose because the values of ρ for Ag^+ ($\rho = 1.0/4.96 = 0.20$) and Tl^+ ($\rho = 0.7/3.56 = 0.20$) are clearly too low (ΔG^s values from [23]).

Reexamination of the results in [6] for TlF yielded a $Y^{0.5}$ extrapolation plot (from 9.2 to 10.3 eV) and $E^t = 8.26$ eV for the normal emission band of Tl^+. The corresponding value $R \approx -1.6$ eV computed for $\Delta G = 2.2$ eV [22] is normal whereas the value $R \approx -0.7$ eV for the autoionization band is anomalous. The threshold energy for the second band of Ag^+ could not be determined because of emission by the anion (ClO_4^-, $E^t = 8.45$ eV [17]) in the range of photon energies in which this band occurs and is expected.

5. Correlation with thermal electron transfer

The activation free energy $\Delta G_x^{\neq}$ for thermal electron transfer in solution can be correlated [14] to the reorganization free energy R for emission on the basis of the theories [1] of Marcus [8] and Hush [9]. One has $\Delta G_x^{\neq} = w + R_x/4$ in the case in which the change of free energy is equal to zero (isotope labeling). There, w is the work required to bring the two reactants from infinity in solution to the activated complex, and R_x is the reorganization free energy for thermal electron exchange. The latter is [14] $R_x \sim R + R(in)$, where R has its normal value.

Unwitting use of anomalous values of R leads to $\Delta G_x^{\neq}$'s which are too low. This was the case in [14] for $Fe(CN)_6^{4-}$. Using R = -0.6 eV (Table 1), R(in) = -0.38 eV [13], w = 0.04 eV [14], one computes $\Delta G_x^{\neq}$ = 0.29 eV against 0.47 eV from experiment. The value $\Delta G_x^{\neq}$ = 0.56 eV computed from the normal value R = -1.7 eV is closer to the experimental result.

Acknowledgment

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Table 1

Data on cyanometalate complexes

	E^t a) (eV)	ΔG b) (eV)	$-R$ (eV)
$Mn(CN)_6^{4-}$	ca. 4.8	-0.22	ca. 0.5
	ca. 6		ca. 1.7
$Fe(CN)_6^{4-}$	5.5	0.36	0.6
	ca. 6.6		ca. 1.7
$Fe(CN)_5NH_3^{3-}$	5.4	0.37	0.5
$Fe(CN)_5H_2O^{3-}$	5.4	0.49	0.4
$W(CN)_8^{4-}$	5.4	0.57	0.3
$Mo(CN)_8^{4-}$	5.6	0.73	0.4

a) Extrapolation plots prepared from data in [11]. E^t = 6.6 eV for $Fe(CN)_6^{4-}$ from 1.1 eV interval between emission bands in [10].

b) From [12].

Table 2

Correlation between reorganization and solvation free energies

	E^{t} a) (eV)	ΔG b) (eV)	$-R$ (eV)	$-\Delta G^{s}$ c) (eV)	$z^2R/\Delta G^{s}$
OH^-	8.45	1.9	2.05	4.2	0.49
Cl^-	8.81	2.55	1.76	3.30	0.53
Br^-	8.05	2.0	1.55	3.00	0.52
I^-	7.19	1.4	1.29	2.61	0.49
V^{2+}	6.38	-0.25	2.13	19.12	0.45
Cr^{2+}	6.14	-0.41	2.05	19.28	0.43
Mn^{2+}	8.08	1.56	2.02	18.92	0.43
Fe^{2+}	7.85	0.77	2.08	19.58	0.42
Co^{2+}	8.60	1.84	2.26	20.85	0.43

a) From [6,7].

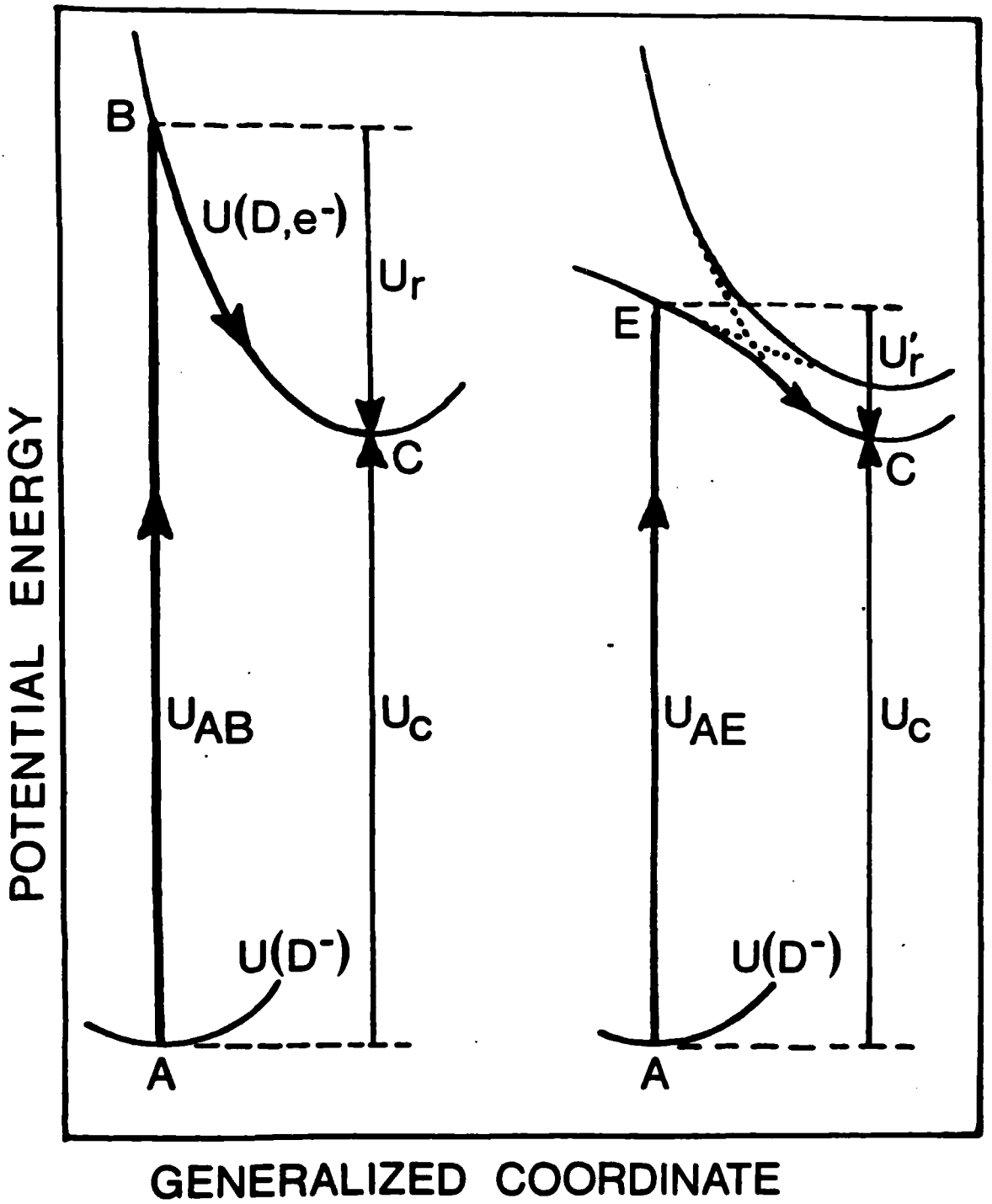
b) From [12,18].

c) From [23] except for OH^- [7].

Captions to Figures

Fig. 1. Potential energy against generalized coordinate for nuclear configuration in the cases of direct optical transition to the continuum (left) and autoionization (right).

Fig. 2. Plots of the emission yield Y to the powers 0.4 (top) and 0.5 (bottom) against photon energy for 1 M $NaNO_3$ (data from [17]).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U(D,e⁻)
Ur
C
E
U'r
C
POTENTIAL ENERGY
UAB
UC
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UC
U(D⁻)
U(D⁻)
A
A
GENERALIZED COORDINATE

FIG. 1

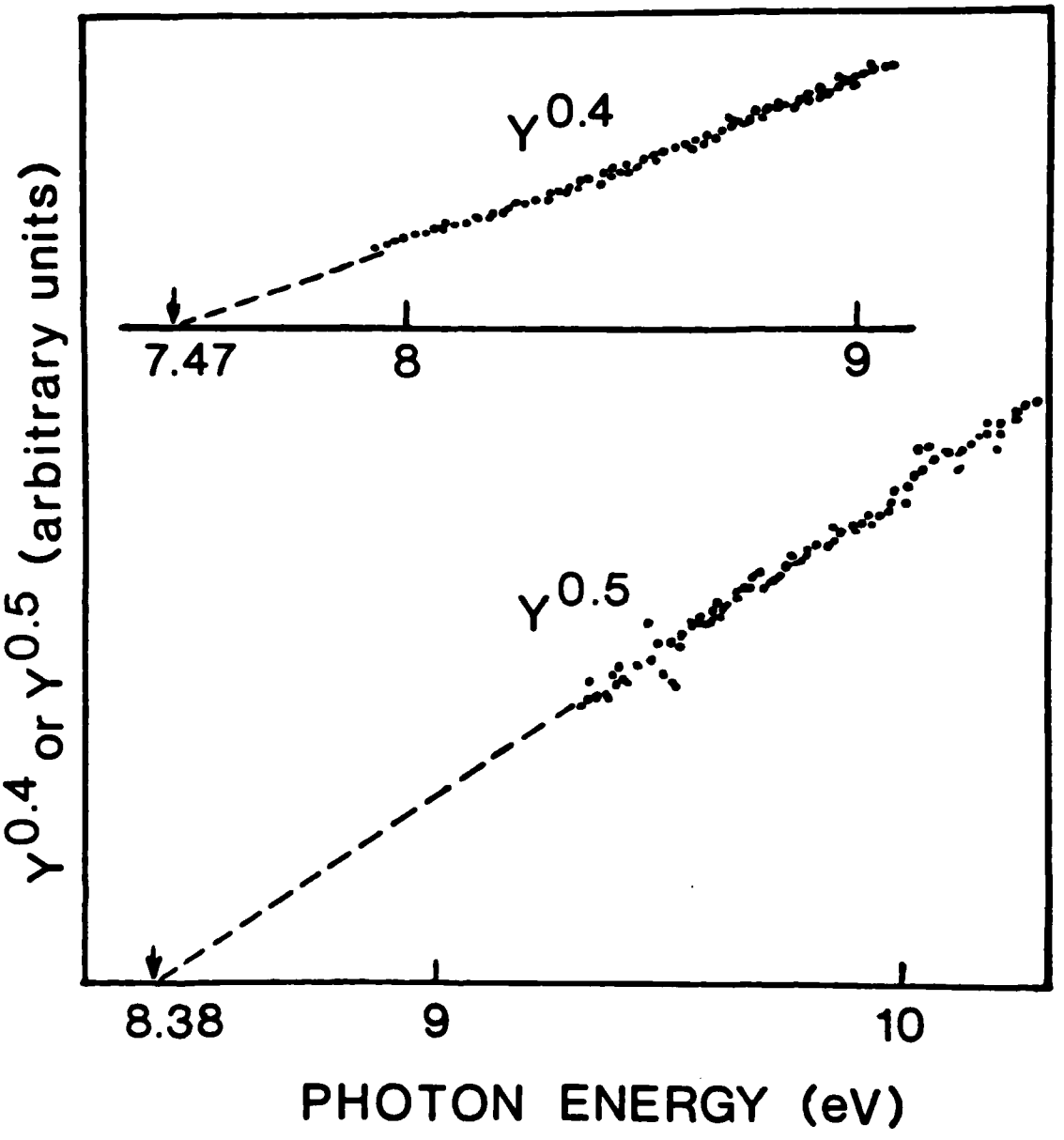
$Y^{0.4}$
7.47
8
9
$Y^{0.5}$
$Y^{0.4}$ or $Y^{0.5}$ (arbitrary units)
8.38
9
10
PHOTON ENERGY (eV)

FIG. 2

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